

Final Report

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Project Title: *Smart Processing of Biodiesel and Fuel Ethanol on Michigan Farms*

Executive Summary

As the price of crude oil continues to rise, the impact on the Michigan Farmer is a reality that is keenly felt. Farm income is associated with a tight margin, and fuel prices are a major factor in whether this margin will allow economic survival of the farm. To address this issue, we evaluated the potential application of a farm-scale production of fuel ethanol from sugar beet, the production of biodiesel from common oilcrops and the use of fuel ethanol, instead of methanol to produce the biodiesel. As the project progressed, some factors became obvious. First, it does not seem economical for a farmer to crush his own beets, when the long-established European method of using diffusion sugar can be applied. It is more economical for a farmer to bring beets to the plant and buying back (less beet cost) raw diffusion sugar. This provides additional revenue for the plant and offers the farmer economy of scale not available to him otherwise. Second, the transport and brewing of the juice is best done in a single large tank that has been sterilized with the juice present prior to brewing. Brewing in barrels is fraught with potential contamination problems, rendering lower yields, hence higher per gallon fuel cost. As with any process, economy of scale plays an important part in distillation. Thus, we recommend against a small scale pot still of the type we employed and recommend a cooperative effort to purchase a stripper still with a rectifying column. Biodiesel production, with fuel ethanol, is likewise most economically carried out on sufficient scale that the residual glycerin byproduct can be sold for an additional profit. The small quantities of glycerin made in a home production unit is generally insufficient to interest a commercial buyer and becomes a waste product that must be disposed. It is recommended that the processes be carried out in a building that can be warmed by the low quality waste heat, to get secondary usage of this particular waste.

Introduction to the Project

Michigan is a significant importer of fossil fuels to support its home heating, transport, manufacturing and farming sectors. State-produced fossil energy supplies less than 25% of Michigan's energy needs, and regional biomass contributions to the state's energy profile are sufficiently negligible as to be unnoticed. Importation of fuels moves operating and investment capital out of the state, makes the state energy supply vulnerable to the whims of the commodities markets, and places the state in an unenviable position with regard to energy security.

Michigan is a major agricultural supplier to the world, with crops that currently meet animal feed and consumer food commodities markets. The intent of this project is to illustrate a practical method for the Michigan farmer to diversify markets through the production of fuel ethanol and biodiesel from oilseeds and sugar beets. We propose to build a pilot plant that could

be replicated at many different scales ranging from the single farm level to possibly the level of a regional cooperative. The intent is to show a scaleable model, providing a tangible means to stabilize and elevate crop prices while securing the state's energy and capital positions.

The overall scope of the project was to provide a demonstration of how a farmer can produce his own tractor fuel (ethanol and biodiesel) with a small farm system. As our results indicate, economy of scale for a small processor may provide an unfavorable energy return. The project consisted of four tasks. The first task was to show the steps involved in production and processing of ethanol from beet juice. The second was to demonstrate the process of production of biodiesel on a small scale. The third task was to show how the first two processes can be merged to generate biodiesel using the ethanol produced from beets. The fourth task was to demonstrate the use of the fuels and tools in a greenhouse setting.

Task 1. Fuel ethanol from sugar beets

Our initial proposal indicated that we would examine two methods of obtaining beers from beet: crushing followed by direct brewing and the brewing of diffusion juice. This portion of the proposal was changed to align more closely with the European model. We used two protocols that are slight modifications of the proposed process. The first was to use the “dirty” juice from the sliced cossettes directly after they emerge from the diffusion extractor. Thus, we reduce the amount of insoluble materials in the beer production. This, in turn, removes the need to do a costly centrifugation step. Since this is to make a replicable model, any centrifugation step could steeply increase the capital cost of a plant beyond reasonable. Thus, a farmer could bring beets to the processing plant and buy back juice from the processing plant as a cost-saving measure. The second step we used was to obtain raw juice after clarification by lime treatment. This gives a juice that has fewer particulates. Two hundred gallons of the first cossette extraction juice was taken as equivalent to the juice of one ton of beets. One hundred gallons of the second clarified raw juice was used as stated. Further, since the extraction process uses very hot water, chemical sterilization was not used. This decision may have had an adverse impact on yield later on (*vida infra*).

After reviewing commercially available distillation units and consulting with a distillation expert (Mr. Lanny Robbins of Larcoman, LLC), we decided it would be most economical and appropriate to the mission of this project to build an ethanol still in the university engineering laboratory. The approach to build our own still allowed us to consider availability of components for the small farm operator as well as scalability for larger operations. Consequently, we designed an inexpensive distillation system using low-cost plumbing supplies available from an ordinary hardware store. During the second quarter, we completed the still (see Figure 2 below), tested the heating controls and prepared standard operating procedures. For our research purposes we added two thermocouples to monitor boiler temperature and distillate temperature in real time.

In February of 2007, the distillers permit was finally obtained from the BATF enabling immediate work on this task. A scanned copy of this permit was included at the end of the fifth quarter report. A discussion of the long delay in obtaining the distillation permit is outlined in a later section.

Four 50-gallon barrels of raw sugar beet diffusion juice were obtained from the Michigan Sugar plant in Bay City, Michigan on March 16. Rather than use whole beets and crushing them at SVSU, we opted to have the beets sliced at the plant and the juice extracted by diffusion, as explained above. On March 19, two 50-gallons of processed, clarified juice were obtained from the plant. The sugar content in both cases was 20% based on the technician's oral report.

After sitting for 3 days indoors to allow the juice to cool to room temperature, each barrel was treated with four ounces of a brewing salts mixture (magnesium sulfate, ammonium sulfate) and two packets of standard wine yeast. Both were obtained from a local winemaking shop (Begick's Greenhouses of Bay City). Bubble traps were set into the lids, and the yeast conversion was allowed to proceed for two weeks. Figure 1 shows the six barrels of sugar beet juice undergoing fermentation.

After settling for an additional week, the solutions were stabilized by addition of 300 ml of a commercial 10% quaternary amine sterilant (QuatSan®, Gordon Food Service of Saginaw). This prevents the secondary growth of bacteria and fungi in the beers that may have occurred during yeast introduction and handling. QuatSan® was chosen because it is nonvolatile and will not introduce toxic fumes into the atmosphere, unlike bisulfite, azide or cyanide salts. Laboratory analysis at completion of fermentation showed alcohol levels to be approximately 5-10% by volume. Yields in some barrels (expected at 10% for a 20% sugar) were half of expected (5%). It was determined that these barrels had gone "sour" because of an acetic acid odor and a low pH. This may have been a consequence of not chemically sterilizing material after collection and prior to yeast addition, or a result of acetate-producing organisms from the air entering the drum at the time of yeast addition. In either case, it was apparent that a sulfur dioxide sterilization in a single large vat followed by yeast addition would be a wise modification to the process. The use of multiple drums opens the door to more facile contamination of beers.



Figure 1. Fermentation vessels for sugar beet juice.

With fermentation complete, the beer was distilled at intervals for production of high-proof alcohol. Some of the beers were sacrificed for evaluating the conditions of distillation. Thus, the distillation process initially proceeded cautiously while proper temperature control settings were found which yielded the most efficient distillation rate. This optimum rate is about 0.026 gal (100 mL) of product per hour, any faster and the percentage of ethanol in the product decreases. Higher distillation rates would require a larger distillation column in order to yield similar purity levels. This in turn, would require a larger distillation pot to compensate for the extra vapor volume in the system. A review of the yields from the distillation showed marginal recovery of ethanol from the beers. This pot still, although inexpensive to make, was a false economy. A better still would have been a stripper still with a rectifying column to obtain 95% ethanol directly. Part of the problem with the inexpensive still is that the heater element was at the midpoint of the volume and thus did not get an efficient conversion of ethanol to vapor. An additional weakness of the still is that its element corroded and failed toward the end of the process. With this, the final barrel did not get processed.

The ethanol still can process up to 10 gallons of beer per run. Each run has been yielding about 0.7 gallon (2.7 L) of an 80% ethanol/20% water product. When enough of this product has been collected, it was intended to be passed through for a second distillation which should yield about 95% ethanol. However, this step has been well documented, and our experience with this particular pot still is that it would have had a poor recovery overall due to element location. Figure 2 shows the ethanol still in operation. Table 1 shows the output of the still prior to failure.



Figure 2. Ethanol still in operation.

Table 1. Still Records for Ethanol Recovery

Attempt #	Barrel #	Stillhead Temp °C	% EtOH	Amount Collected ml	Pure EtOH (ml)	Gal	Overall yield
1	1	80.0	76	160	121.6		
2	1	80.0	85	237	201.5		
3	1	80.0	50	702	351.0		
4	1	85.0	77	212	163.2		
5	1	85.0	80	150	120.0		
6	1	91.0	56	252	141.1		
7	1	89.0	68	230	156.4		
8	1	84.0	80	212	169.6		
9	1	84.0	78	197	153.7		
10	1	90.0	82	403	330.5		
11	1	84.0	60	50	30.0		
12	1	84.0	80	1000	800.0		
13	1	84.0	80	1050	840.0		
14	1	84.0	82	2340	1918.8		
15	1	84.0	84	1800	1512.0		
				Sum	7009.3	1.8	3.6%
16	2	83.1	86	1640	1410.4		
17	2	85.0	81	2250	1822.5		
18	2	85.0	84	2090	1755.6		
19	2	87.0	84	2030	1705.2		
20	2	84.0	83	2080	1726.4		
				Sum	8420.1	2.2	4.4%
21	3	80.0	81	1200	972.0		
22	3	82.0	81	2660	2154.6		
23	3	82.0	83	1915	1589.5		
24	3	80.0	82	2100	1722.0		
25	3	90.0	74	2510	1857.4		
				Sum	8295.5	2.2	4.4%

Task 2. Biodiesel from regional agriculture-derived oilsThe oilseed press:

To demonstrate a complete process for fuel processing, we purchased components for an oilseed press and subsequently assembled the unit on a steel frame attached to a flatbed automobile trailer for portability. The complete oilseed press consisted of a screw-type oilseed press (Model 6YL-80, China Double Elephants Machinery Corporation, China), driven by a 12 horsepower diesel engine (Model S195M, Changfa Corporation, China) and a 7.5 kilowatt alternating current electric alternator.

We performed a series of laboratory experiments to measure the capability of the oilseed press to produce soybean oil. In these experiments, measured amounts of three varieties of soybean seeds were poured into the pre-warmed oilseed press, and the expelled oil was collected

and measured. The left-over soybean press cake was then taken to the laboratory where any remaining oil was extracted with hexane. This experiment gave information on oilseed press operation, seed variety, and fuel efficiency. Additionally, fuel consumption was measured with the engine operating on 100% biodiesel that was obtained from local sources.

For each experiment, a level 5 gallon bucket of soybeans (0.54 bushels, 32 pounds) was used. Complete processing of each 5 gallon sample required 20 minutes. The three varieties of soybeans were Renwick Soybean (a high oil, Roundup ready seed), Royster Clark (Roundup ready), and Marque Secan (a typical non genetically modified soybean seed). The results for each variety are given below.

Sample A, Renwick Soybean (roundup ready)

Volume: 2.112 L oil (or 0.557 gallons) from 0.54 bushel: *1.0 gallons oil/bushel soybeans*

*Mass: 1943 grams (or 4.28 pounds) from 32 pounds: *13.4% oil by mass*

Sample B, Royster Clark (roundup ready)

Volume: 1.490 L oil (or 0.393 gallons) from 0.54 bushel: *0.73 gallons/bushel*

*Mass: 1490 grams (or 3.02 pounds) from 32 pounds: *9.4% oil by mass*

Sample C, Marque Secan

Volume: 1.285 L oil (or 0.339 gallons) from 0.54 bushel: *0.63 gallons/bushel*

*Mass: 1182 grams (or 2.60 pounds) from 32 pounds: *8.1% oil by mass*

*Note: This was calculated using the density of soybean oil (0.920 g/mL) and the density of soybeans (60 lbs/bushel). Volumes were measured using volumetric laboratory glassware.

We extracted the remaining oil from the soy meal from above using hexane with the following results:

Sample A yielded 6.02% oil by mass

Sample B yielded 6.12% oil by mass

Sample C yielded 6.62% oil by mass

By adding the percent by mass of the pressed oil and the hexane extracted oil from above, we have a measure of the total oil available for biodiesel production for each variety of soybean. As seen in our results below, different soybean varieties can have widely different oil content. Sample A, the Renwick seeds, were labeled as a high oil seed.

Sample A: 19.4% oil

Sample B: 15.5% oil

Sample C: 14.7% oil

Notable in the results is the consistent amount of oil left in the seed press cake regardless of original oilseed content. This is directly related to the tightness of the oilseed screw press. To show this, we also performed hexane extraction on press cake that had been generated in an earlier experiment where the feed screw in the oilseed press was tightened to a lesser degree than in the experiments above. In that earlier press cake, we recovered 8% oil by mass, which is higher than the 6% values mentioned above. The higher percentage of oil is caused by the

formation of a thicker press cake, a direct result of the lesser degree of feed screw tightening in that earlier experiment.

Each sample required 20 minutes of elapsed time during which the raw soybeans were first poured into the oilseed press and the resulting press cakes were subsequently fed through the oilseed press three times. Exactly one hour of elapsed time occurred during which all of the samples were consecutively processed by this method. During that hour, a total of 4.89 liters (1.29 gallons) of soybean oil were recovered from the oilseed press. Also during that same hour, the engine powering the oilseed press consumed a total of 2.24 liters (0.59 gallons) of biodiesel.



Figure 3. Press cake was fed into the oilseed press a total of three times in order to extract the maximum amount of soybean oil. A hand-held pyrometer was used to measure the temperature of the press cake as it exited the screw press.

The biodiesel processor and wash tank:

There many biodiesel processors listed for sale around the country. Most of them consist of one tank for the reaction and another tank for washing and also include necessary pumps for transferring the liquids. Many of these commercial biodiesel units cost several thousand dollars, yet they can be made with common parts available at local plumbing and agricultural supply stores for only a few hundred dollars. We took the latter approach and assembled a biodiesel reactor.

Important considerations for the biodiesel reactor included availability of components as well as scalability. Accordingly, a biodiesel reactor was assembled using an “Appleseed Processor” design published by Maria Alover (www.biodieselcommunity.org) based on a modified water heater. We used a 25 gallon electric water heater. Modifications to the water heater included bypassing the upper heating element, adding a transfer/mixing pump, and adding piping/fittings necessary to fill, drain, and circulate the contents of the unit. A thermocouple was installed in the biodiesel boiler tank to monitor the temperature.

Separate from the processor, we assembled a wash tank consisting of an agricultural “induction tank” available at agricultural equipment dealers that supply sprayer supplies. The tank is semi-transparent to allow the operator to see the biodiesel and water layers that will form during the washing process. The tank was assembled with a drain valve in the bottom, and a series of fine spray water nozzles in the lid, and the necessary fittings to use water from an outdoor water faucet.



Figure 4. Pictured are the washing tank and the biodiesel processor. The second picture shows a clip from the Saginaw News.

Biodiesel was made from both methanol and ethanol in the laboratory. The product from each was indistinguishable with respect to physical properties (viscosity, density, color), and the methods to make each are very similar. Due to the low yields of ethanol from the brewing and distillation process described earlier, it was necessary to purchase methanol locally for biodiesel production. Methyl ester biodiesel was made using soybean oil, corn oil, and canola oil from local sources which included both used cooking oils as well as pure oil.

In the 25 gallon biodiesel processor, several small batches of biodiesel were made using 70 lbs of oil (approximately 10 gallons), 14 lbs of methanol (approximately 2 gallons), and about 1.0 lb of KOH. Additional KOH was needed for the used cooking oil, this was determined using a titration to determine the free fatty acids on the oil. The procedure began by transferring the oil into the biodiesel processor and pre-heating to 80°C. While the pre-heating the oil, in a separate container KOH was slowly added to the methanol to prepare the potassium methoxide solution. Of note is the intense heat generated during this process which can cause poisonous and explosive methanol fumes. Once the oil reached 80°C, the potassium methoxide solution was added to the oil in the biodiesel processor, and the pump was used to mix the entire contents for 60 minutes. The heater was then turned off and reaction mixture was allowed react and settle for 24 hours.

During the 24 hour reaction period, the byproduct glycerin (approximately 7 lbs) settled to the bottom of the processor. After 24 hours, the glycerin was drained from the processor and

stored for future fuels research. A sample of biodiesel was removed to check for completeness of reaction with IR and GC. If not complete, time and/or a small amount methoxide was added followed by additional testing. Once reaction was complete and most of the glycerin was removed, the biodiesel was transferred to the wash tank.

In the semi-transparent, cone-bottomed wash tank it was then possible to completely drain any residual glycerin. Then water was by connecting a garden hose through a water timer to the fine spray nozzles in the top of the tank. For a ten gallon batch of biodiesel, three washes of 5 gallons of water were sufficient to remove residual glycerin, methanol, and potassium hydroxide. The final quality of the biodiesel was determined by GC analysis using the ASTM method.

We produced approximately 9.5 gallons of product for every 10 gallons of oil input (a 95% yield). The low yield was realized because some biodiesel was sacrificed during the separation steps after water washing in an attempt to remove extra bound glycerin residues. ASTM specifies maximum levels of free and total glycerin. Water washing is shown to be effective in reducing the free glycerin but not bound glycerides. The presence of bound glycerides indicates an incomplete reaction, though only slightly for the samples shown in Table 2. To reduce bound glycerides, a longer reaction time and/or a larger proportion of alcohol to triglyceride oil has been effective as shown in the results. Figure 5 below shows a typical GC chromatogram of a biodiesel sample.

Table 2: GC analysis of biodiesel			
	Free glycerin	Bound glycerin	Total glycerine (free + bound)
Pre-washed sample, 10% MeOH, 1% KOH	0.26%	0.34%	0.60%
Washed sample, 10% MeOH, 1% KOH	0.02 %	0.34%	0.36%
Washed sample, 20% MeOH, 1%KOH	Not detectable	0.20%	0.20%
ASTM specification	0.02% max	Not specified	0.24% max

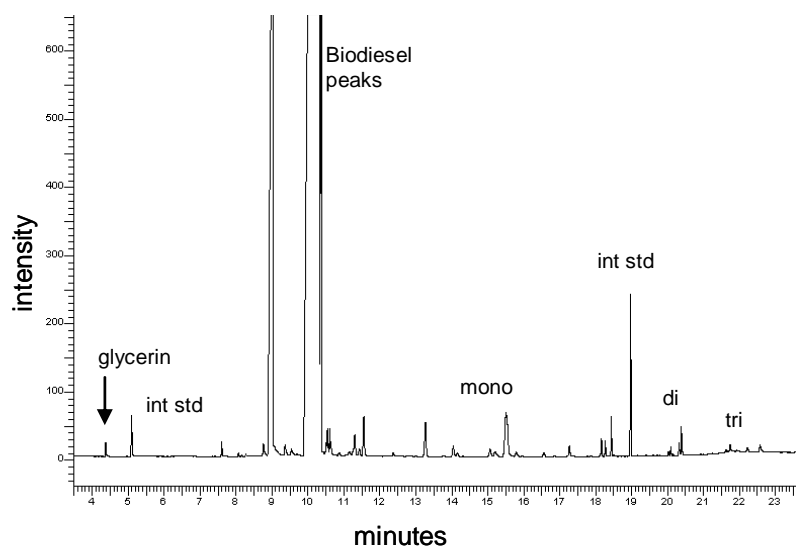


Figure 5. Shown is a GC chromatogram using ASTM method D-6584.

Task 3. Integration of ethanol and biodiesel processing and cogeneration of heat and electricity.

Task 3 was originally intended to be carried out in the greenhouse as a way to provide carbon dioxide during fermentation and waste heat to warm the greenhouse during distillation processes. However, the base heating system was not installed in the greenhouse in sufficient time to allow the fermentation there. In addition, the Bureau of Alcohol, Firearms and Explosives (BATFE), which issued the alcohol permit prohibited the transfer of the equipment to the relatively unsecured greenhouse area. In addition, delays in obtaining a heating plant and electricity for the greenhouse significantly impacted our initial abilities to develop an adequate secure facility outside of Science East Building. Further, delays in the permitting process at BATFE were not resolved until January, after the intervention of Senator Levin's office. These problems revealed to us issues that were not previously obvious. The first is that the initial information supplied to us by BATFE that we obtain an "Experimental Plant Permit" turned out to be incorrect. I was informed by more senior officials at BATFE that an "Alcohol Fuels Plant Permit" would have been easier for their clerks to approve. While a greenhouse can be made sufficiently secure to meet BATFE concerns, we recommend, on the basis of our experience, that the plant be placed in a lockable outbuilding with power and heat already present.

Instead, the integration of the processes was done in the Science East building where the brewing and distillation took place. We were able to demonstrate that ethanol easily transesterifies triglycerides in the presence of base to yield ethyl esters of fatty acids (biodiesel). Our larger scale productions used methanol as a transesterification alcohol in the process. It was available in our laboratory and functions the same as dry ethanol.

Biodiesel prepared in the bioreactor was used to run a Changfa generator attached to an oil press and a generator. The engine worked flawlessly. Ethanol was used in turn to power an alcohol-fueled genset successfully.

Overall, we recommend that small scale operations for ethanol production not be done, because the economy of scale of a larger system would yield better fermentation and recovery. Biodiesel may be made on a small scale, but again, testing small lots for quality according to ASTM standards costs the same for 5 gallons as it does for 1000 gallons, and economy of scale favors the larger batches. Also, larger batches produce glycerin byproduct in volumes that approach interesting market quantities. Small batches produce glycerin waste that must be disposed in sanitary sewer or in composting piles, in which case, they lose the value of a market offset.